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Research Article

Investigations of Electron Properties of Carbon Nanotubes Decorated with Platinum Nanoparticles with Their Varying Fraction

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The paper presents the outcomes of investigations into electron properties and a qualitative and quantitative analysis of the chemical composition of unmodified carbon nanotubes with comparison to nanotubes decorated with platinum nanoparticles. The fabricated nanocomposite materials of the CNT-Pt type differed in a varying mass concentration of platinum nanoparticles (5, 10, and 20% of Pt). X-ray photoelectron spectroscopy (XPS) was employed to examine the structure and to analyse chemical composition. Survey spectra measurements within a wide range of the binding energy of 0–1400 eV were performed in the first phase of the investigations. The core lines of C1s carbon, Pt4f platinum, and O1s oxygen and a valence band were then measured. The chemical composition of the studied materials was determined based on the measured spectra of the core lines. The purpose of the investigations is to determine the effect of platinum nanoparticles on the structure and electron properties of the fabricated CNT-Pt nanocomposites.

1. Introduction

The principal task imposed on the present and future generations of engineers is to design and fabricate new materials with unique properties. The requirements that engineering materials must satisfy these days become out of date very rapidly due to efforts to achieve better and better functional characteristics of the products fabricated with such materials. The discovery of carbon nanotubes in 1991 has launched a series of intensive studies into this new allotrope type of carbon, confirming their unique mechanical, electrical, thermal, and optical properties [1–4]. Carbon nanotubes are characterised by a large specific area and high electrical conductivity. Moreover, they are chemically stable, elastic, resistant to bending, stretching, and torsion, and also lightweight [5–7]. Their density—depending on the type—ranges over 0.6–2.6 g/cm³ [8, 9]. A broad spectrum of nanotubes' uncommon properties indicates their substantial application opportunities as independent materials or components of material

systems. Scientists are especially interested in nanotubes because of their chemical modification, which opens up utterly new application vistas for nanotubes. This new branch of investigations into nanotubes encompasses molecular surface sorption, attachment of function groups to their surface and endings, improved wettability, and also the additions of other elements outside, inside, and within a graphene nanotube plane. The chemical functionalisation of carbon nanotubes enables developing completely new composite materials combining the unique properties of nanotubes and other materials, where it becomes possible to control their electrical, mechanical, thermal, and optical properties [10–15]. The results of own research and the research pursued by research institutions [16–23] confirm that deposition of nanoparticles of noble metals such as Pd, Pt, Rh, and Au onto a surface of carbon nanotubes may contribute to a substantially higher density of carriers of free loads and hence to nanotubes' enhanced thermal and electrical conductivity. A practical application of the so developed

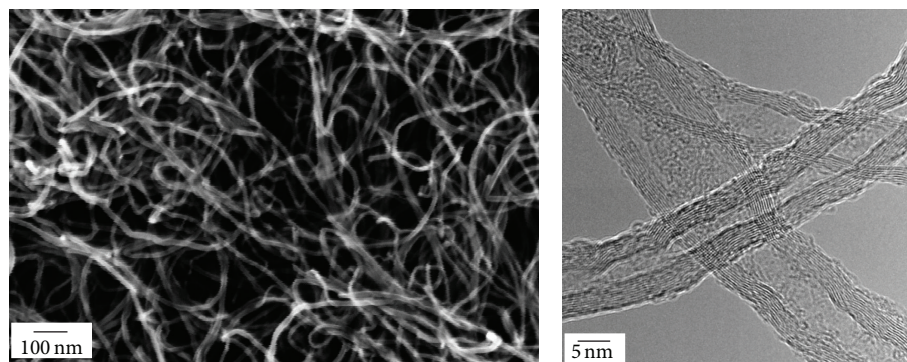


FIGURE 1: SEM and TEM images of multiwalled carbon nanotubes used in the investigations [37].

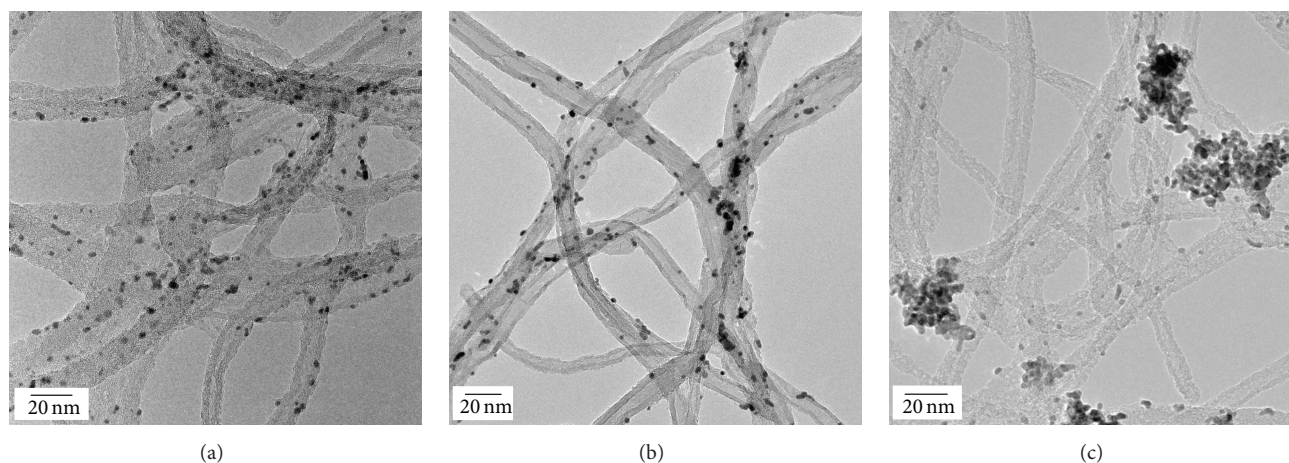


FIGURE 2: TEM photographs of CNT-Pt nanocomposites with varying fraction of Pt nanoparticles: (a) CNT-5% Pt, (b) CNT-10% Pt, and (c) CNT-20% Pt [37].

nanotubes-nanoparticles type materials is sensors of selected chemical substances, including H_2 , CH_4 , and NO_2 [24]. The key advantages of sensors containing active material in the form of a CNT-Pt composite are high chemical and thermal resistance, no requirement to work at an elevated temperature, as in a majority of optional solutions, operating sensitivity, fast reaction, and selectivity.

This paper presents the results of investigations into the structure and electron properties of CNT-Pt type nanocomposite materials with a varying mass fraction of platinum nanoparticles.

2. Experimental

Multiwalled carbon nanotubes with the length of 100–200 μm and diameter of 10–20 nm fabricated with the Chemical Vapour Deposition (CVD) technique were employed for investigations whose aim was to recognise the electron properties of the fabricated nanocomposite materials (Figure 1). 8% chloroplatinic acid, H_2PtCl_6 , supplied by Sigma-Aldrich (USA) was used for synthesising the platinum nanoparticles.

Three sets of specimens containing, respectively, 5, 10, and 20% of platinum nanoparticles deposited on the surface of carbon nanotubes were prepared for spectroscopic studies

(Figure 2). CNT-Pt type nanocomposites were prepared by the preliminary treatment of the surface of carbon nanotubes with function groups ($-COOH$, $-COH$, and $=CO$) through their covalent functionalisation in a mixture of acids. Platinum nanoparticles were synthesised by way of chemical reduction in the second phase of the process, and then they were deposited onto the surface of nanotubes. A methodology of carbon nanotubes' functionalisation and decoration with platinum nanoparticles was already detailed in the authors' earlier works [19, 24]. The investigations of physiochemical properties together with a qualitative and quantitative analysis of chemical composition of unmodified carbon nanotubes and CNT-Pt type nanocomposites were performed with X-ray photoelectron spectroscopy (XPS) using a PHI 5700/660 photoelectron spectrometer by Physical Electronics. Monochromatic X-ray radiation coming from an AlK_{α} aluminium anode with the radiation energy of $h\nu = 1486.6$ eV was used to carry out the measurements. The energy resolution of the spectrometer used for the tests was 0.3 eV.

The material intended for the tests was deposited directly onto the surface of a PREVAC PTS STANDARD specimen carrier and placed in an XPS measuring chamber. Survey spectra were measured, within a wide range of binding energy

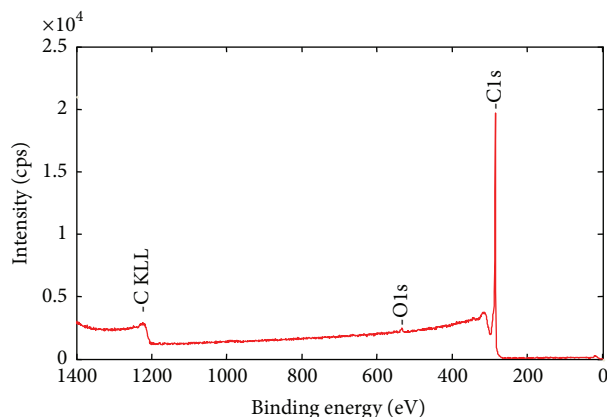


FIGURE 3: XPS spectrum of unmodified carbon nanotubes.

of 0–1400 eV. The core lines of C1s carbon, Pt4f platinum, and O1s oxygen and a valence band were then measured. The measurements were made in a high-resolution mode with pass energy of 23.5 eV. The chemical composition of the studied materials was determined on the basis of the measured spectra of core lines and MultiPak software was used for chemical composition calculations [25]. The shape of the core lines was determined with the Voigt profile containing a compound Doniach-Sunjc function [26–28]. The Shirley function was used for determination of background lines [29, 30].

3. Results and Discussion

Figure 3 shows a review spectrum created as a result of examining unmodified carbon nanotubes with X-ray photoelectron spectroscopy excited with X-ray photoelectron spectroscopy. Two main photoemission lines are shown on the spectrum for which the maximum counts for the binding energy of 285 eV and 533 eV correspond, respectively, to electron states of carbon marked as C1s and oxygen marked as O1s. Carbon content in the nanotube material was identified at the level of 98.7% in atomic terms, while oxygen content is 1.3% in atomic terms. A small amount of the adsorbed oxygen, as compared to the amount of carbon, signifies a good quality of the studied material. Figure 4 shows the measured core lines for the states C1s and O1s obtained in a high-resolution mode. The performed profile analysis of the lines reveals a complex character of carbon lines (Figure 4(a)). A spectrum shape of the C1s line exhibits the presence of three components. The binding energy of the particular components is, respectively, 284.2 eV, 284.4 eV, and 285.9 eV. The energy location of the profile lines corresponds to carbon states with the hybridisation sp^2 and sp^3 in a carbon nanotube [31]. The third component is probably connected with the presence of carbon in the adsorbed CO molecules on the surface of nanotubes [31] or with manufacturing conditions. The O1s line (Figure 4(b)) consists of only one component with the binding energy of about 533 eV. The binding energy of the oxygen line corresponds to OH groups [32]. A wide line for energy above 1200 eV, visible in Figure 3,

corresponds to Auger electron emission as a result of passing between KLL levels for carbon.

Figure 5 presents a valence band achieved for the studied nanotube material. A valence band obtained from the surface of Highly Oriented Pyrolytic Graphite (HOPG) was added for reference. The maximum photoemission of the valence band occurs in both cases for the binding energy of approx. 18.5 eV. An additional feature is visible on the spectrum for both measured specimens at the energy of about 13.5 eV. The third characteristic feature, less visible for CNT material, exists for the binding energy of about 8 eV. A shape of a valence band of CNT material is therefore reflecting a hexagonal structure of carbon bonds in graphite. An interesting valence band area, presenting the electron states related to the existence of π -type and σ -type bonds between carbon atoms in a nanotube, occurs in the binding energy area below 10 eV. The same type of bonds exists also in the electron structure of graphite [33].

Figures 6, 7, and 8 show the results of photoemission measurements of carbon nanotubes modified with platinum nanoparticles (CNT-5% Pt, CNT-10% Pt, and CNT-20% Pt), obtained in a wide range of binding energy. Photoemission lines are visible on the spectra coming from the states of core lines of C1s carbon (about 284 eV), O1s oxygen (about 531 eV), Auger C KLL lines (approx. 1200 eV), and O KLL lines (about 980 eV). Photoemission lines of 4p, 4d, and 4f platinum are visible in the form of *doublets* related to the *spin-orbit* interaction. Photoemission lines related to the states of fluorine, F1s, and sodium, Na1s, occur additionally on the spectrum created for the CNT-10% Pt specimen. Depending on the mass fraction of platinum (5, 10, and 20%) in the newly created nanocomposites, the intensity of photoemission lines for electron states 4p, 4d, 4f, and 5p of platinum is growing.

Figure 9 presents C1s photoemission lines achieved for carbon nanotubes modified with platinum nanoparticles as compared to unmodified nanotubes. Similarly to an unmodified CNT material, a profile analysis of a C1s core line of specimens enriched with platinum was undertaken. Similarly to the input material, the presence of components corresponding to sp^3 and sp^2 hybridisation was demonstrated. A negligible increase in the intensity of the sp^2 component and a decrease in the intensity of the sp^3 component were seen in the modified material versus the CNT input material. The ratio of bonds between carbon atoms, determined for a material modified with platinum nanoparticles according to variations in intensities of sp^3 and sp^2 components, is not changing. It can therefore be assumed that an increased concentration of platinum in the modified material is not substantially changing the number of sp^3 and sp^2 bonds in carbon nanotubes.

Figure 10 shows the 4f core lines of platinum obtained for carbon nanotubes modified with platinum nanoparticles with their varying fraction (5, 10, and 20% by mass of Pt). The structure of the measured 4f states consists of two $4f_{7/2}$ and $4f_{5/2}$ lines connected with *spin-orbit* interaction, creating a so-called *doublet*. A detailed profile analysis has shown the presence of two doublets which moved in relation to each other by 3.3 eV. The first doublet for the binding energy of 71.2 eV for the state $4f_{7/2}$ corresponds to the presence of

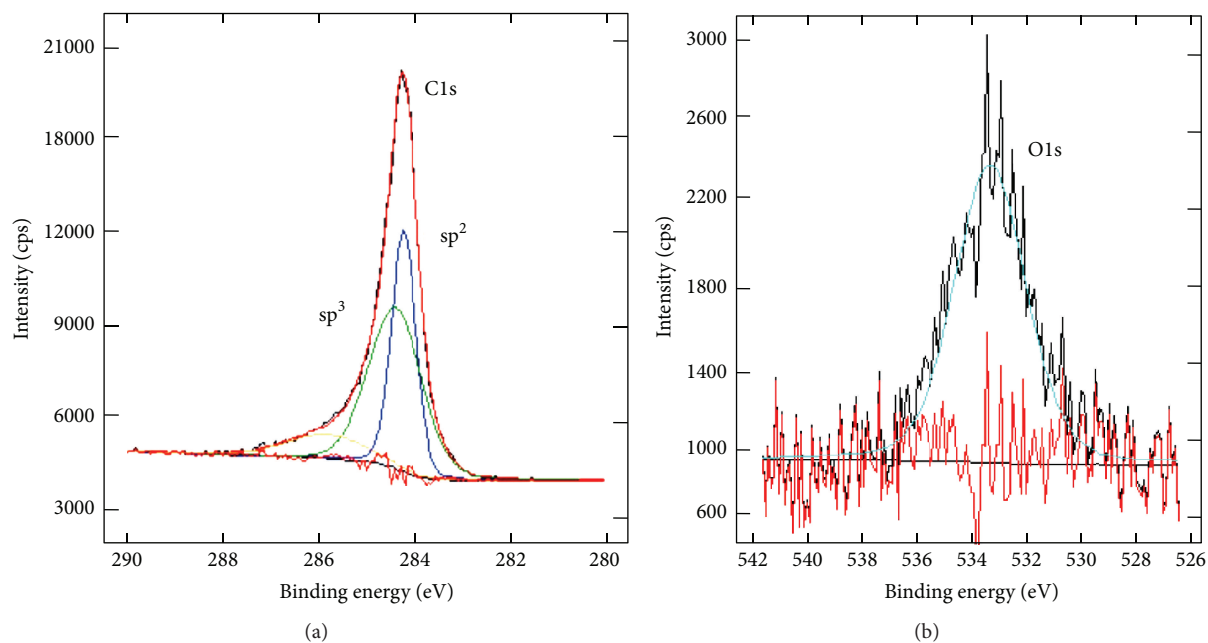


FIGURE 4: Spectra of C1s and O1s core lines obtained in high-resolution mode: (a) deconvolution of C1s line and (b) O1s photoemission line.

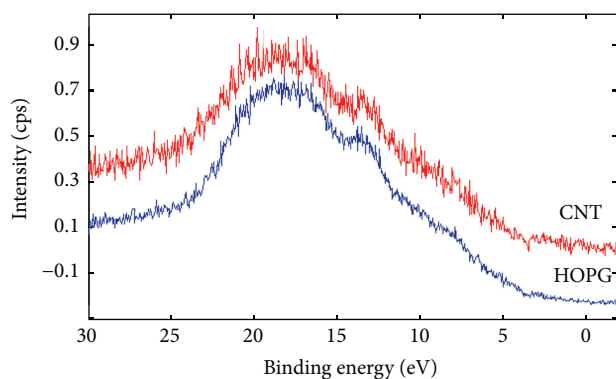


FIGURE 5: Valence band of unmodified nanotubes of CNT and graphite HOPG.

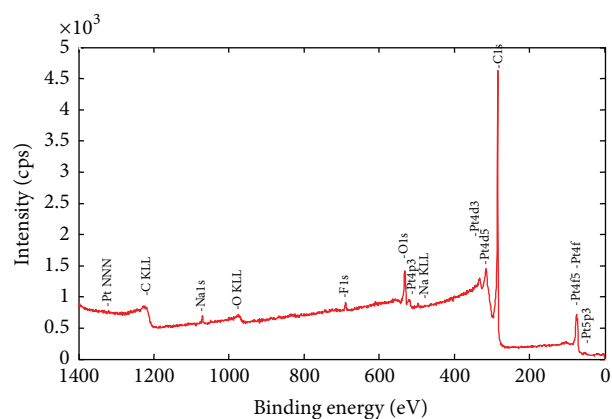


FIGURE 7: XPS spectrum of carbon nanotubes modified with CNT-10% Pt platinum nanoparticles.

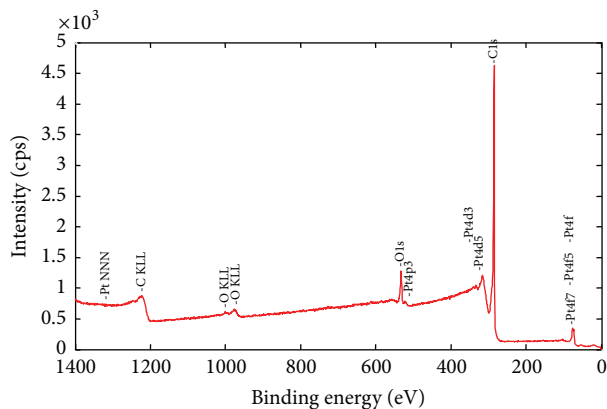


FIGURE 6: XPS spectrum of carbon nanotubes modified with CNT-5% Pt platinum nanoparticles.

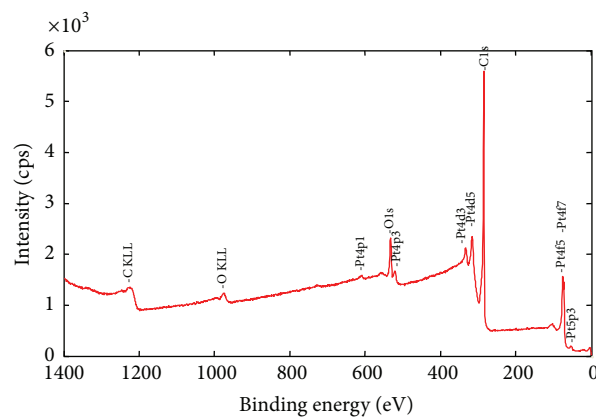


FIGURE 8: XPS spectrum of carbon nanotubes modified with CNT-20% Pt platinum nanoparticles.

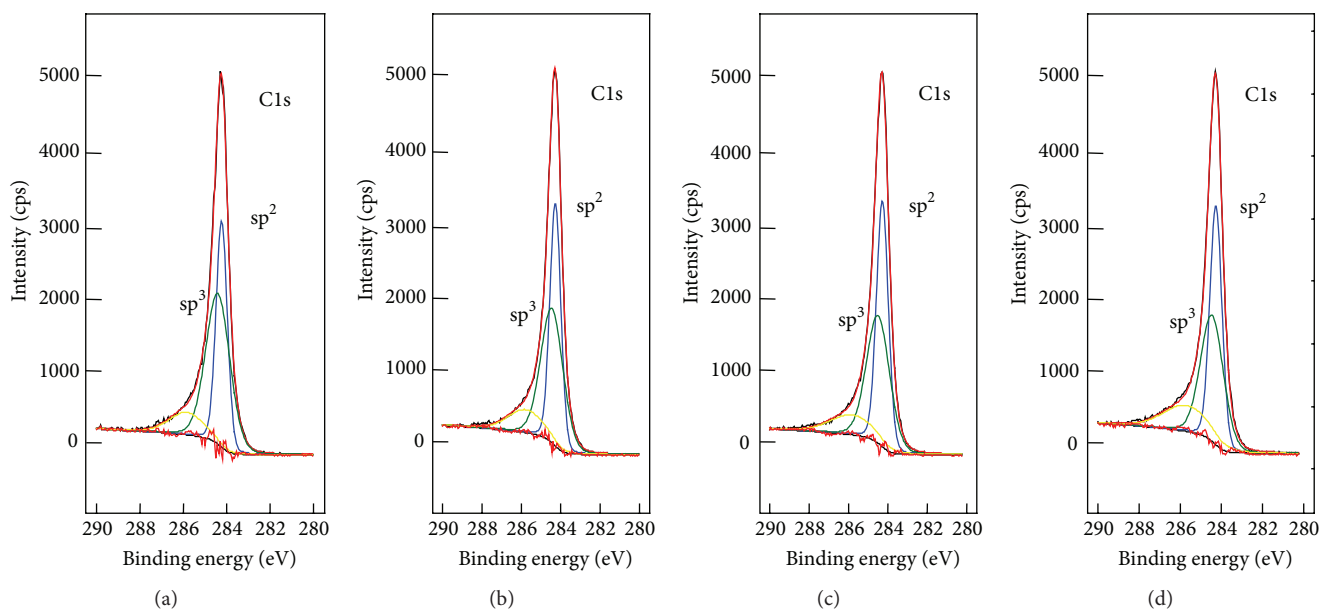


FIGURE 9: Spectra of C1s core line for (a) unmodified carbon nanotubes (CNT) and nanotubes modified with platinum nanoparticles for their varied mass fraction, including (b) CNT-5% Pt, (c) CNT-10% Pt, and (d) CNT-20% Pt.

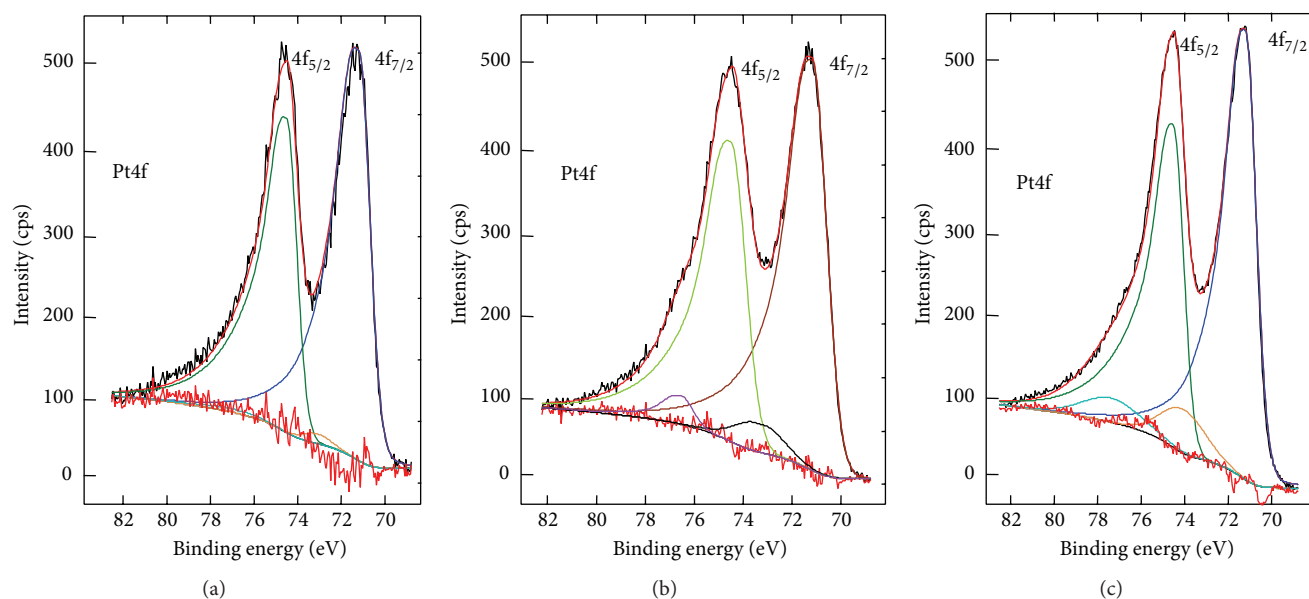


FIGURE 10: Spectra of Pt4f core line for carbon nanotubes modified with platinum nanoparticles for their varied fraction, including (a) CNT-5% Pt, (b) CNT-10% Pt, and (c) CNT-20% Pt.

platinum in a metallic state, while the other doublet for the binding energy of 74.5 eV for the state $4f_{5/2}$ is connected with platinum oxide PtO_2 [31, 34–36]. It is apparent from Figure 10 that the intensity of the doublet $4f_{5/2}$ is rising as the percentage fraction of platinum is growing.

Figure 11 shows the measured core lines for the states O1s of oxygen obtained in a high-resolution mode. The performed profile analysis of the lines reveals a complex character of O1s lines (Figures 11 and 12). A spectrum shape exhibits the presence of five components. The binding

energy of the oxygen line component of 530.2 eV corresponds to platinum oxide PtO_2 (platinum with the 4th level of oxidation of PtO_2). When analysing the intensity of the oxygen line component of 530.2 eV together with a varying mass fraction of platinum of, respectively, 0, 5, 10, and 20% of Pt, growth for this value can be seen (Figure 11). This phenomenon confirms the growth of the platinum oxide, PtO_2 , fraction in the analysed materials along with a varying fraction of nanoparticles (5, 10, and 20% by mass of Pt). The energy location of the successive components of the line O1s

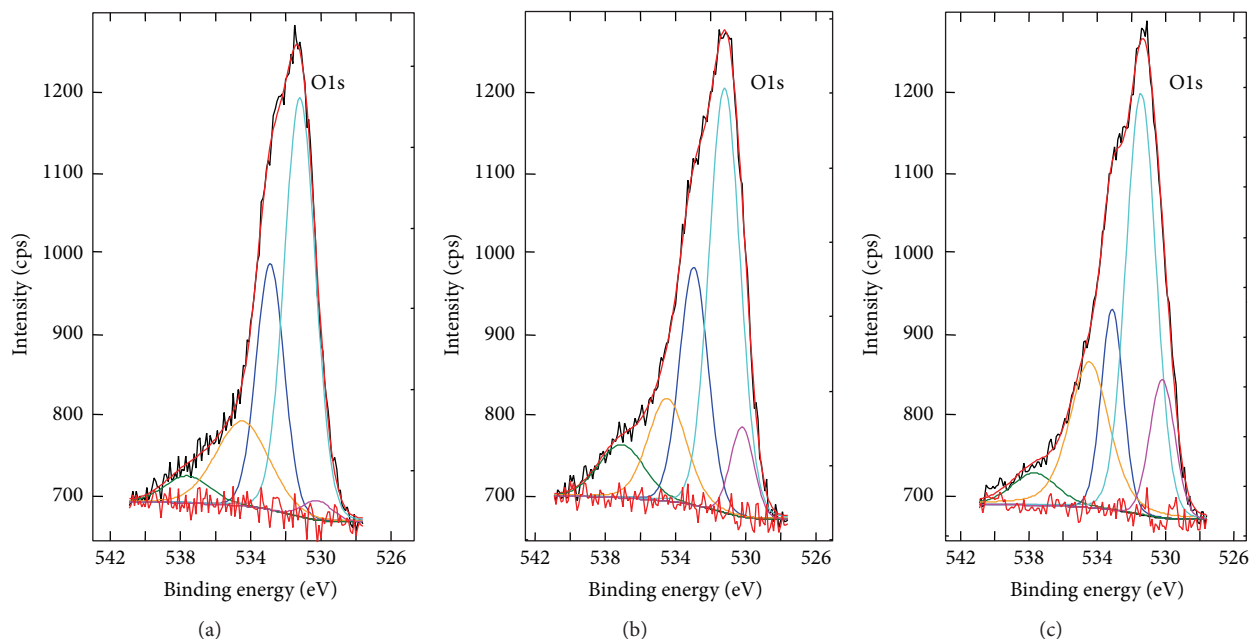


FIGURE 11: Spectra of O1s core line for carbon nanotubes modified with platinum nanoparticles for their varied fraction: (a) CNT-5% Pt, (b) CNT-10% Pt, and (c) CNT-20% Pt.

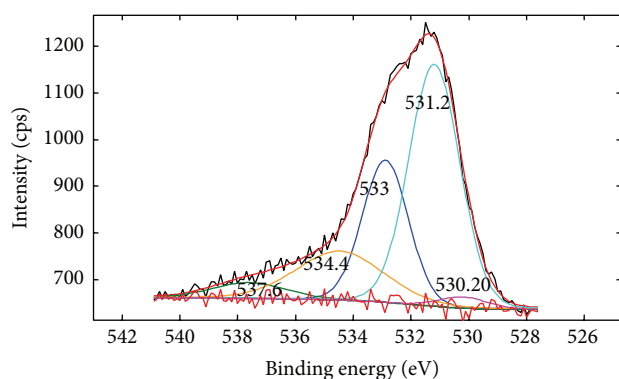


FIGURE 12: Location of O1s line components for carbon nanotubes modified with platinum nanoparticles (CNT-5% Pt).

is shown in Figure 12. The additional oxygen line components signify the presence of oxygen compounds created in the process of carbon nanotubes' modification with platinum.

Figure 13 presents the structure of a valence band of CNT material with a varied degree of modification with platinum nanoparticles as compared to the band of unmodified nanotubes and HOPG graphite. A shape of a valence band for graphite (HOPG) in the lower part of the figure was added for reference. A shape of a valence band of CNT material is therefore similar to HOPG and reflects a hexagonal structure of carbon bonds in graphite. The modification of carbon nanotubes with platinum nanoparticles will deliver 5d platinum electrons to a valence band structure of the nanotubes. Figure 13 allows observing an increase in the photoemission of 5d electrons of platinum depending on the modification degree of the nanotubes with platinum

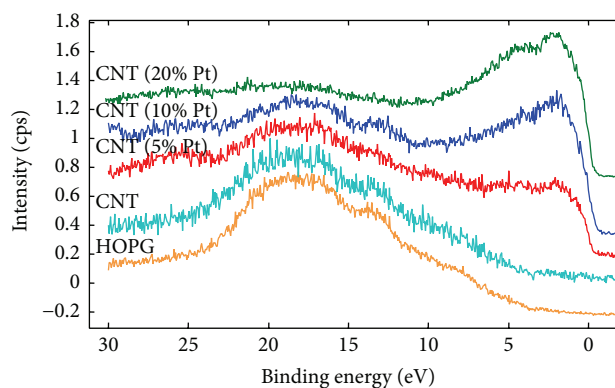


FIGURE 13: Structure of valence band area of carbon nanotubes modified with platinum nanoparticles for their varied fraction of CNT-5% Pt, CNT-10% Pt, and CNT-20% Pt against unmodified nanotubes and graphite.

nanoparticles. The investigations carried out with a standard source of XPS radiation do not allow pointing out variations in the character of π and σ bonds in the analysed materials, due to a high density of 5d state of platinum.

A quantitative chemical composition analysis confirms the higher contents of platinum in the studied nanocomposites along with its varying mass fraction (Table 1). A CNT-5% Pt specimen features the concentration of carbon, oxygen, and platinum with the following concentrations: C (90.9% at.), O₂ (8.2% at.), and Pt (0.9% at.). The chemical composition of the CNT-10% Pt specimen is as follows: C, 90.4% at., O₂, 7.7% at., and Pt, 1.9% at.; and, according to the initial assumptions, platinum content in the analysed materials grows two times (1.9% at.). A twofold growth of

TABLE 1: Quantitative chemical composition analysis of unmodified carbon nanotubes and nanocomposites with varying fraction of platinum (CNT-5% Pt, CNT-10% Pt, and CNT-20% Pt).

Material	C (% at.)	O ₂ (% at.)	Pt (% at.)	Na (% at.)	F (% at.)
Unmodified CNTs	98.7	1.3	—	—	—
CNT-5% Pt	90.9	8.2	0.9	—	—
CNT-10% Pt	90.4	7.7	1.9	<1	<1
CNT-20% Pt	86.5	9.7	3.8	—	—

platinum content of 3.8% at. compared to the CNT-10% Pt specimen was recorded for the CNT-20% Pt nanocomposite with the following % at. composition: C, 86.5%, O₂, 9.7%, and Pt, 3.8%.

4. Conclusion

The results of photoemission measurements (XPS) obtained for the nanocomposites have shown the presence of photoemission lines in the spectra coming from the states of core lines of C1s carbon, O1s oxygen, and 4p, 4d, and 4f platinum, which are visible in the form of *doublets* related to the spin-orbit interaction. As the mass fraction of platinum nanoparticles (5, 10, and 20%) in a CNT-Pt nanocomposite is rising, the intensity of photoemission lines for the electron states 4p, 4d, and 4f of platinum is rising. A profile analysis of a core line C1s of the input material and of this modified with platinum nanoparticles shows the presence of components corresponding to sp³ and sp² carbon hybridisation. It has been confirmed on the basis of the investigations performed that an increased concentration of platinum in the modified material is not substantially changing the number of sp³ and sp² bonds in carbon nanotubes. The modification of carbon nanotubes with platinum nanoparticles delivers, however, 5d platinum electrons to a valence band structure of the nanotubes, which is manifested by a higher density of electron states at the Fermi surface.

Competing Interests

The authors declare that they have no competing interests.

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